Oxide Thickness Measurement by Scanning Electron Microscopy with Controlling Ultra-Low Voltage *

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The secondary electron (SE) images were recorded for Si wafers with and without SiO₂ thin (10 nm-50 nm) films by a scanning electron microscope (SEM) with changing primary electron energy from 2.5 keV down to 0.2 keV during one frame scan. Brightness of the SE image increased up to critical values and then decreased with decreasing primary electron energy for the specimens with SiO₂ films. The critical voltage is higher for specimen with thicker film. These results are discussed by means of the oxide film thickness, the penetration depth of incident electrons, and the surface potential caused by charging. [DOI: 10.1380/ejsnt.2008.35]

Keywords: Scanning electron microscope (SEM); Secondary electron emission measurement; Surface electron phenomena (surface potential); Thin oxides; Silicon oxides

I. INTRODUCTION

Sputter-depth-profiling using surface analysis techniques such as Auger electron microscopy (AES) and Secondary ion mass spectrometry (SIMS) have been practically used for measuring thickness of surface thin layers on a local area. These techniques are time-consuming especially when one obtains two-dimensional mapping of the film thickness.

Scanning electron microscopy (SEM) is one of the most popular techniques to investigate sample surfaces in many fields of science and engineering. SEM has been widely used to obtain two-dimensional information of surface, such as topology and the average atomic number. We have investigated material surfaces using ultra-low-voltage SEM. The distribution of thin oxide layers on steel sheet has been clearly seen as a dark contrast in secondary electron (SE) images using low primary electron energy (E_p) [1, 2]. The origin of the dark contrast has been explained by the following effect [2]; When the penetration of primary electron beam is limited within the oxide layer, the surface of the oxide layer is charged by the electron beam irradiation. The surface potential caused by the charging has positive sign and is limited only few eV when the E_p is less than about 1 keV-2 keV because the secondary electron yield is higher than 1 [3]. This charging reduces the emission yield of secondary electrons when kinetic energy of secondary electrons are smaller than the surface potential. This scenario provides us with a possibility of estimating thickness of thin oxide films with low electric conductivity by means of the comparison between the contrasts of SE images recorded with different E_p , i.e. different penetration depth of primary electrons. In this paper, we will present preliminary results for SiO₂ films, in which the SE images were recorded with controlling E_p

including ultra low energy less than 1 keV.

II. EXPERIMENTAL

The SE images were recorded for SiO₂ films with the thicknesses of 10 nm, 19 nm, and 50 nm on Si wafers by a Schottky Emission SEM model LEO 1530 (LEO, now Carl Zeiss, Germany). As a reference, Si wafer etched by HF was also observed. The E_p was changed from 2.5 keV down to 0.2 keV (0.1 keV step) during one frame scan. The primary electron beam was from the direction of the surface normal. Typical beam current is 0.16 nA at $E_p = 1$ keV. Scan area is about $180\mu\text{m} \times 135\mu\text{m}$ and scan speed of primary electron beam was about 0.6 msec/ μm . The in-lens type SE detector which has a high sensitivity to the electrons with low kinetic energy [4] was used. The SE images were recorded digitally and average brightness of SE images was measured for area corresponding to each E_p using an image-processing software.

Variable parameters of the objective lens, such as focus and stigmation, and the detector as well as the position of specimens including the working distance were adjusted at the starting E_p . During a frame scan, these parameters were untouched but only the E_p was changed. Therefore, it should be noted that the fixed parameters were not optimized for other E_p 's except for 2.5 keV.

III. RESULTS

Figure 1 shows SE images for Si-wafer with (a) 50 nm-SiO₂, (b) 19 nm-SiO₂, and (c) Si wafer etched by HF, in which E_p was changed from 2.5 keV to 0.2 keV with a step of 0.1 keV. The SE images are divided into areas recorded with different E_p by the horizontal lines corresponding to time when E_p was changed. The top part of each image was recorded with E_p of 2.5 keV and the bottom part was recorded with E_p of 0.2 keV. For the HF-etched Si wafer, another image was recorded (the insert in Fig. 1(c)) with a reduced gain of image because the brightness of the SE image was saturated at low E_p as shown in Fig. 1(c). The

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FIG. 1: SE images for Si-wafers with (a) 50 nm-SiO₂, (b) 19 nm-SiO₂, and (c) Si wafer etched by HF, in which E_p was changed from 2.5 keV to 0.2 keV (step is 0.1 keV). The insert in figure (c) is SE image recorded with a reducing gain of the detector.

SE image becomes brighter with decreasing E_p down to 0.2 keV for the Si wafer etched by HF. For the specimens with SiO₂ films, with the E_p decreases, the SE images become brighter at high E_p as well as for the HF-etched Si wafer. However images become dark at intermediate E_p 's and then become bright again with decreasing E_p .

The average brightness of SE image were estimated for each area recorded with each E_p by a 8 bits scale. Figure 2 (a) shows the average brightness as a function of E_p . Figure 2 (b) shows the normalized data as the brightnesses in the E_p rang from 1.5 keV to 2.5 eV becomes the same level. Here, we define that the E_p where the brightness of the SE image for the specimens with SiO₂ films starts to decrease is a critical primary energy (E_{pc}). The E_{pc} determined as E_p where the brightness starts to deviate from curves for the HF-etched Si wafer are about 1.3 keV, 0.7 keV, and 0.3 keV for SiO₂ films with the thickness of 50 nm, 19 nm, and 10 nm, respectively. It is obviously that the E_{pc} is higher for specimen with thicker SiO₂ film.

FIG. 2: (a) Average brightness of SE images as a function of E_p . (b) Normalized average brightness as the values becomes almost same in the E_p range from 1.5 to 2.5 eV. The roughly estimated critical values (E_{pc}) are denoted by arrows for the specimens with SiO₂ films.

IV. DISCUSSION

The dependence of the SE yield on E_p for Si was reported [5, 6], in which the SE yield increases monotonically with E_p decreases down to about 0.3 keV-0.5 keV. No significant changes of the SE yield have been reported at intermediate E_p between 0.3 keV and at least 10 keV. The E_p dependence of the brightness of SE image for the HF-etched Si (in Fig. 2) is similar to these phenomena of the reported SE yield. We consider that the change of the brightness of SE image relates to the SE yield and can be used as a reference curve of a smooth change of SE yield.

The SE yield of SiO₂ increases monotonically as E_p decreases down to about 0.2 keV from 1 keV as well as that for Si [5]. The brightness of the SE image for SiO₂ films changes at the E_{pc} higher than 0.5 keV and the E_{pc} changes with thickness of SiO₂ films as shown in Fig. 2. These results indicate that the E_{pc} relates to the thickness of SiO₂ films. The effective penetration range of electrons (R_p), which is the distance over which the electrons travel and deposit their excess energy, was estimated using an empirical relationship, $R_p \sim 1000E^{1.4}$ [7]. The penetration depth is also estimated from results of a Monte Carlo Simulation. These results are shown in Fig. 3 as a function of E_p . The E_p 's where these values are close to the thick-

FIG. 3: Attenuation range of electrons as a function of E_p . Estimation of the values is mentioned in the text.

ness of each oxides (0.3 keV-0.5 keV for 10 nm, 0.7 keV-0.8 keV for 19 nm, and 1.1 keV-1.5 keV for 50 nm-SiO₂) are similar to the E_{pc} (about 0.3 keV, 0.7 keV, and 1.3 keV, respectively). This result strongly suggests that the E_{pc} corresponds to the E_p which the penetration of the incident electrons reaches to the interface between the SiO₂ films and Si substrates. The total SE yield for SiO₂ films are larger than 1 at the E_p range lower than at least 3 keV [8]. For the E_p lower than E_{pc} , the SE yield is reduced by the positive surface potential caused by the charging because the penetration of primary electrons is limited within the insulating SiO₂ layers. Therefore, as mentioned in the introduction, the SE image becomes darker due to the positive charging in this situation [2]. When the E_p exceeds the E_{pc} , the primary electrons reach to the Si substrate. The surface potential becomes equal or near to that of the substrate and therefore hardly restricts the SE emission.

Our results demonstrate the possibility that one can estimate the thickness of SiO₂ film using SEM by evalu-

ating the E_{pc} and the effective penetration range. This technique can be extended to the surface layers composed by the materials with low electric conductivity. The E_{pc} is determined by monitoring the change of the brightness of SE with changing E_p as shown in Fig. 2. However, the changes of brightness around the E_{pc} are always not sharp but are broad transition in our results. This may be due to many contributions; the distribution and density of primary electrons in the films, the transport properties of electrons between the surface and the substrate. The broad transition makes the determination of the E_{pc} difficult. The investigations of theoretical and experimental approaches are highly required for quantitative estimation of thickness of thin surface layer using SEM. From experimental point of view, the optimization of SEM parameters is needed to controlling surface charging. It is also expected that the system that allow direct SE intensity measurement will be developed in the future.

V. CONCLUDING REMARKS

We proposed a method to estimate thickness of thin surface layers with low electric conductivity using SEM and demonstrated the possibility by presenting the results for thin SiO₂ films. This technique uses the penetration depth of the primary electrons as shallow as the thickness of surface layer (less than few tens nanometers). It should be emphasized that the low E_p is one of key points because the typical E_{pc} is 0.3-1.3 keV for SiO₂ films with the thickness of 10 nm-50 nm.

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